Perfluoro Acid Anhydrides

Product Specification

Trifluoroacetic acid anhydride (TFAA), pentafluoropropionic acid anhydride (PFPA), and heptafluorobutyric acid anhydride (HFBA) are perfluoroacylated acylation reagents. These reagents form stable, volatile derivatives with alcohols, amines, and phenols. The major use for perfluoro acid anhydrides is to prepare electron-capturing derivatives for GC/electron capture detection. This detector provides greatly enhanced responses for halogenated derivatives. If reaction times and temperatures are adjusted to allow for the differences in reactivity, the similarities among TFAA, PFPA, and HFBA are sufficient to allow these reagents to be interchangeable.

Although the anhydride may be used on its own, the acylation reactions go most smoothly and quickly in a solvent and with a catalyst. Bases such as triethylamine (TEA) and trimethylamine (TMA) often are added to promote reactivity. PFPA and HFBA should be used with an acid scavenger, to help drive the reaction to completion and to prevent column damage from acidic byproducts of the derivatization reaction.

TFAA is the most reactive and most volatile of the three anhydrides. No acid byproducts are formed in derivatization reactions with TFAA. Amino acids and steroids are the analytes most commonly derivatized with TFAA. Amino acids are derivatized primarily to obtain volatile derivatives, not for monitoring with an ECD.

PFPA is used to prepare volatile derivatives of alcohols, amines, and phenols for use with either an ECD or an FID. PFPA should be used with an acid scavenger to prevent column damage. The derivatives require a low analysis temperature.

HFBA provides the derivatives most sensitive to ECD. Like PFPA, it yields volatile derivatives of alcohols, amines, and phenols for ECD or FID, and should be used with an acid scavenger to prevent column damage.

Features/Benefits

Produce stable, volatile derivatives of alcohols, amines, and phenols for electron capture or flame ionization detection.

Frequently used in confirmation testing for drugs of abuse by GC/MS. (TFAA is used to identify methamphetamine, PFAA is used to identify opiates and benzoylecgonine, HFBA is used to identify amphetamines and phencyclidine.)

Typical Procedure

This procedure is intended to be a guideline and may be adapted as necessary to meet the needs of a specific application. Always take proper safety precautions when using an acetylating reagent – consult MSDS for specific handling information.

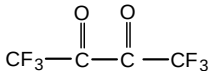
Prepare a reagent blank (all components, solvents, etc., *except sample*), following the same procedure as used for the sample.

1. Dissolve 50µg sample (250µg for FID) in 0.5mL benzene.
2. Add 0.1mL 0.05M trimethylamine (acid scavenger) in benzene, followed by 10µL PFPA, HFBA, or TFAA.
3. Cap the vial and heat at 50°C for 15 minutes.
4. Cool the mix and add 1mL 5% ammonia in water.
5. Shake for 5 minutes, allow the layers to separate, and inject an aliquot of the benzene (upper) layer onto the chromatograph.

Properties

Trifluoroacetic Acid Anhydride

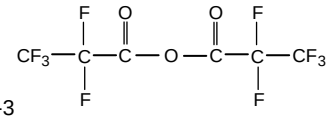
Structure:



CAS Number: 407-25-0
Molecular Formula: $(CF_3CO)_2O$
Formula Weight: 210.03
bp: 39.5-40°C
d: 1.487
n_D: <1.300
Appearance: clear, colorless liquid

Pentafluoropropionic Acid Anhydride

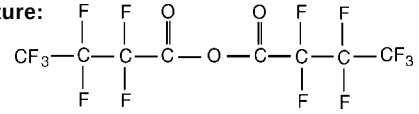
Structure:



CAS Number: 356-42-3
Molecular Formula: $(CF_3CF_2CO)_2O$
Formula Weight: 310.05
bp: 69-70°C
d: 1.571
n_D: <1.300
Appearance: clear, colorless liquid

Heptafluorobutyric Acid Anhydride

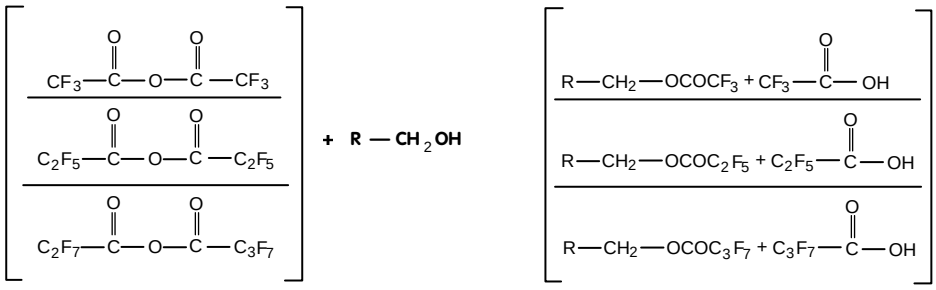
Structure:



CAS Number: 336-59-4
Molecular Formula: $(CF_3CF_2CF_2CO)_2O$
Formula Weight: 410.06
mp: -43°
d: 1.665
n_D: 1.2870
Appearance: clear, colorless liquid

797-0244,0245,0246

Derivatization times vary widely, depending upon the specific compound(s) being derivatized. If derivatization is not complete under the procedure described here, the addition of a catalyst, use of another solvent, higher reaction temperature, longer reaction time, and/or higher reagent concentration should be evaluated.



797-0399

Mechanism (1,2)

Acylation involves the introduction of an acyl group into a molecule that has a replaceable hydrogen atom (OH, NH, or SH group). **Except for TFAA,** anhydride acylating reagents form acidic byproducts that must be removed prior to GC analysis, to prevent destructive effects on the phase in the column. Consequently, acylations with anhydride reagents normally are performed in pyridine, tetrahydrofuran, or another solvent capable of accepting the acid byproducts.

Toxicity – Hazards – Storage – Stability

Perfluoroacyl anhydrides are corrosive, flammable, and moisture-sensitive. Store in a bottle or ampul at room temperature, in a dry, well ventilated area. Use only in a well ventilated area and keep away from ignition sources. Moisture can hinder the reaction.

Recommended storage conditions for the unopened product are stated on the label. If you store an opened container or transfer the contents to another container for later reuse, validate that your storage conditions adequately protected the reagent.

References

1. K. Blau and J. Halket *Handbook of Derivatives for Chromatography* (2nd ed.) John Wiley & Sons, New York, 1993.
2. D.R. Knapp *Handbook of Analytical Derivatization Reactions* John Wiley & Sons, New York, 1979.

Additional Reading

F.F. Lawrence and J.J. Ryan, J. Chromatogr. 130, 97 (1977).
D.E. Coffin, J. Assoc. Off. Anal. Chem. 52, 1044 (1969).
N.P. Sen, J. Food Sci. 34, 22 (1969).
D.D. Clarke, *et al.,* J. Gas Chromatogr. 5, 307 (1967).

Ordering Information

Description	Cat. No.
TFAA	
10 x 1mL	**3165-U**
25mL	**33164**
PFPA	
10 x 1mL	**33167**
25mL	**33168**
HFBA	
10 x 1mL	**33170-U**
Microreaction Vessels with Hole Caps and Septa	
1mL, pk. of 12	**33293**
3mL, pk. of 12	**33297**
5mL, pk. of 12	**33299**
Books	
Handbook of Derivatives for Chromatography K. Blau and J. Halket	**Z246220**
Handbook of Analytical Derivatization Reactions D.R. Knapp	**23561**

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